Operation of a Third Generation JPL Electronic Nose in the Regenerative ECLSS Module Simulator at MSFC

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An electronic nose has been developed at the Jet Propulsion Laboratory (JPL) to monitor spacecraft cabin air for anomalous events such as leaks and spills of solvents, coolants or other fluids with near-real-time analysis. It is designed to operate in the environment of the US Lab on ISS and was deployed on the International Space Station for a seven-month experiment in 2008-2009. In order improve understanding of ENose response to crew activities, an ENose was installed in the Regenerative ECLSS Module Simulator (REMS) at Marshall Space Flight Center (MSFC) for several months. The REMS chamber is operated with continuous analysis of the air for presence and concentration of CO, CO_2, ethane, ethanol and methane. ENose responses were analyzed and correlated with logged activities and air analyses in the REMS.

Nomenclature

ECLSS	=	Environmental Control and Life Support System
ENose	=	Electronic Nose
EXPRESS	=	EXpedite the PRocessing of Experiments to Space Station
FTIR	=	Fourier Transform Infrared Spectroscopy
JPL	=	Jet Propulsion Laboratory
ISS	=	International Space Station
MSFC	=	Marshall Space Flight Center
ppm	=	parts-per-million
REMS	=	Regenerative ECLSS Module Simulator

I. Introduction

An electronic nose has been developed at the Jet Propulsion Laboratory (JPL) to monitor spacecraft cabin air for anomalous events such as leaks and spills of solvents, coolants or other fluids with near-real-time analysis. The Third Generation ENose is designed to detect, identify and quantify eight common organic species and three inorganic species, ammonia, mercury and sulfur dioxide.

The Third Generation ENose has been designed to operate in the environment of the US Lab on ISS. It was deployed on the International Space Station for a seven-month experiment in 2008-2009. An impediment to full

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understanding of ENose response onboard ISS is the lack of fully characterized analyses of the air during the operational period. In order to gain a better understanding of ENose response to crew activities, an ENose identical to that used on ISS was installed in the Regenerative ECLSS Module Simulator (REMS) at Marshall Space Flight Center (MSFC) for several months while volunteer crew members exercised, cooked food and simulated other daily activities.

II. Installation of ENose in the REMS

A. The Regenerative ECLSS Module Simulator

The REMS is a human-in-the-loop chamber designed to study the capabilities of air and water recycling systems used in human-occupied spacecraft. It is a chamber of approximately 200 m^3 which uses a cabin air assembly similar to that on ISS, with a flight-like heat exchanger. It operates at ambient pressure for its location, slightly under 1 atmosphere at MSFC, and at a temperature of 20 – 22°C. The REMS has been used to study the performance of water recovery systems with a recent focus on water and other chemical species evolved during crew activities[1]. During the time that ENose was installed in the REMS, performance of the new vapor-phase photocatalytic reactor was under study. In this study, an atmospheric contaminant load was generated and collected inside the REMS. Human volunteers performed exercise and hygiene activities simulating crew activities on-orbit to generate wastewater, adhering to tested protocols developed and fine-tuned for past water recovery tests. Additionally, a contaminant injection system was used introduce volatile contaminants into the REMS atmosphere to more accurately mimic a space-based environment. The REMS chamber is operated with continuous analysis of the air for the presence and concentration of five species, CO, CO_2, ethane, ethanol and methane. Chamber air was analyzed using in-line Fourier Transform Infrared Spectroscopy (FTIR) on the recirculating air. One analysis was done each five minutes. Concentration of the five analyzed species is recorded as parts-per-million.

All activities were logged and described; the most frequent activity was exercise. Volunteers used exercise equipment and logged time and weight change during the exercise period; weight change was considered to be caused by water lost to the REMS environment. Exercise equipment includes four treadmills, two elliptical trainers and one stationary bicycle. In addition to exercise, volunteers used facility water to perform hygiene activities such as tooth brushing, washing and shaving, and heated frozen meals in a microwave oven[2].

B. The JPL Electronic Nose

An electronic nose has been developed at the Jet Propulsion Laboratory (JPL) to monitor spacecraft cabin air for anomalous events such as leaks and spills of solvents, coolants or other fluids with near-real-time analysis. It is an array–based sensing system which is designed to run continuously and to monitor for the presence of selected chemical species in the air at concentrations related to the 24 hour Spacecraft Maximum Allowable Concentration (SMAC), generally several parts-per-million (ppm) for organic and high parts-per-billion (ppb) for inorganic species. The JPL ENose uses an array of 32 semi-selective chemical sensors; sensing materials are primarily polymer-carbon composite films, but also include inorganic films and carbon nanotubes[3,4].

There have been three phases of development of the JPL Electronic Nose. In the first phase, a device capable of detecting, analyzing and quantifying ten analytes at the 1-hour SMAC was developed. This device was tested successfully in 1998 on Space Shuttle flight STS-95[5]. In the second phase, the ENose was miniaturized and the capabilities were significantly expanded to include 21 analytes and detection at varying humidity and temperature. The Second Generation ENose was tested extensively on the ground and was demonstrated to be able to detect, identify and quantify the 21 analytes at or below their 24-hour SMACs[6]. The Third Generation ENose, shown in Figure 1, was built as a Technology Demonstration instrument and was operated for seven months aboard the International Space Station (ISS). This device has a volume of 3.6 L and has a mass of 3.4 kg. Its power requirement is 12-15 W average and 20 W peak. The Third Generation ENose was trained to detect, identify and

Figure 1. The Third Generation ENose. *The Sensor Unit, developed as the 2nd Generation ENose, is enclosed in the Interface Unit, which connects to an EXPRESS Rack on ISS.*

quantify releases of eleven selected chemical species at ranges related to the 24 hour SMAC[7] for each species. The eleven species and their quantification targets are shown in Table 1, below. ENose analysis was designed to report the concentration of targets chemical species at 1/3 to 3 times the quantification target; concentrations are expressed as parts-per-million (ppm) at 1 atmosphere.

Table 1: Analyte List and Detection Target Concentrations for ENose Technology Demonstration.

	ANALYTE	QUANT. TARGET (ppm)	REASON TO DETECT THIS SPECIES	24 HOUR SMAC (ppm)
Inorganic	Ammonia	5.0	Thermal control system external coolant; potential leak into cabin	20
	Mercury	0.010	High profile; used in ISS Hg vapor lamps and certain payloads	0.0020
	Sulfur Dioxide	1.0	Thionyl chloride battery leakage potential	no SMAC
Organic	Acetone	200	Frequently detected in ISS atmosphere	200
	Dichloromethane	10	Always detected in ISS atmosphere	35
	Ethanol	500	Frequently detected in ISS atmosphere; ECLSS concern	2000
	Formaldehyde	0.10	Prevalent off gas product; health concern; allergen sensitivity	0.10
	Freon 218	20	Russian A/C coolant; leaks have occurred; ECLSS concern	11,000
	Methanol	10	Frequently detected in ISS atmosphere	10
	2-Propanol	100	Frequently detected in ISS atmosphere; ECLSS concern	100
	Toluene	16	Represents aromatic compounds; frequently detected	16

The results of the data analysis for the Technology Demonstration on ISS have been discussed in detail elsewhere[8]. Briefly, several events of methanol, formaldehyde and Freon 218 were detected; most events lasted 30 to 60 minutes, with the longest event about 2 hours. There was a cyclic change in relative humidity, which was reliably correlated to operation of the Carbon Dioxide Removal Assembly. There was also repeated change in humidity which correlated in time with scheduled crew exercise periods. None of the chemical release events could be correlated in time with operation of any specific instruments or with any crew activities. There was a repeated occurrence of an unknown chemical species; this repeated occurrence could not be correlated in time with any scheduled activities. Identification of that species is the subject of another paper in this conference[9].

C. ENose Experiment Set-up in the REMS

The purpose of the experiment with ENose in the REMS was to determine how the ENose would respond in an environment similar to that of the ISS, in particular to determine how the ENose sensing array would respond to exercise, food preparation, and hygiene activities, and whether such activities would result in false positives and/or detection of unknown species. The experiment in the REMS allowed us to correlate ENose response to particular activities and to understand how the sensor array and analysis software would respond to and interpret regular activities. Such data were taken on ISS during the seven-month technology demonstration, but we did not have access to logs of as-performed activities, time and duration of activities, location of activities and other information which could be used in interpreting ENose data, although we did use schedules to attempt to correlate events.

The ENose was set up in the REMS in the forward section of the chamber, near an air uptake vent. By locating ENose near an air uptake vent, we maximized the possibility that any chemical species out-gassed, generated or injected into the chamber would pass by the ENose inlet and be detected by the sensing array.

The ENose operated continuously in the REMS for several months, from April – August, 2009. During the period of operation, data were downloaded from the ENose to JPL for analysis. While ENose was operating, the

REMS was operated as planned, with volunteers providing contaminant load and with periodic injections of selected chemical species, and with continuous analysis of the chamber air for the presence of CO, CO_2, ethane, ethanol and methane as well as relative humidity and temperature. This paper will focus only on results from ENose operation; results of ECLSS-related testing and activities will be the subject of other reports. A photograph of the interior of the REMS with people exercising is shown on the left side of Figure 2; the ENose in the REMS is shown on the right side of Figure 2 (circled). In the view of the chamber, ENose is not in the picture; it is located in front of and toward the wall from the person on the right of the picture.

Figure 2. The REMS and ENose in the REMS. *Volunteers exercise in the REMS(left) to create contaminant load; ENose (circled, on right) operated continuously near the entry door of the REMS to detect targeted contaminants listed in Table 1t.*

III. Results and Discussion

The ENose operated continuously in the REMS from early April through August, 2009. ENose sensor data are taken continuously, with three points per minute, and a single file of sensor data is created for each day from midnight to midnight. These files were downloaded once a week for analysis at JPL. After the end of the test period, logs of activities in the REMS as well as FTIR analysis of the chamber air were transferred to JPL for correlation of activities with ENose responses.

Initial analysis of ENose data showed that the primary response of ENose sensors was to changes in water content, or relative humidity, in the air. The analysis software returned very few events of targeted species and occasional events classified as unknown.

Two weeks of operation were selected for detailed analysis. The weeks selected were April 27, 2009 through May 8, 2009. These two weeks were selected as representative of exercise and other atmospheric load activities, and completed logs for the volunteer crew members were available for these days. No activities were undertaken on weekends, so a selection of two weeks gives ten active and four quiet days. During the inactive days, the chamber was kept closed, there were no entries or exits, no exercise, and no injections were made. During the active days, crew members entered the chamber through an air lock, and exercised or engaged in other activities. On some of the active days ethanol was injected in order to study the ability of the ECLSS to remove it from the air.

Examples of data taken on a weekend day are shown in Figure 3. On the left side, FTIR data show little change in the concentration of methane, a metabolic product, which is consistent with no activity on this day. The FTIR data show a moderate change in concentration of CO_2, possibly owing to the ECLSS system under test. On the right side, ENose sensors showed no changes in environment, and there was little fluctuation in humidity.

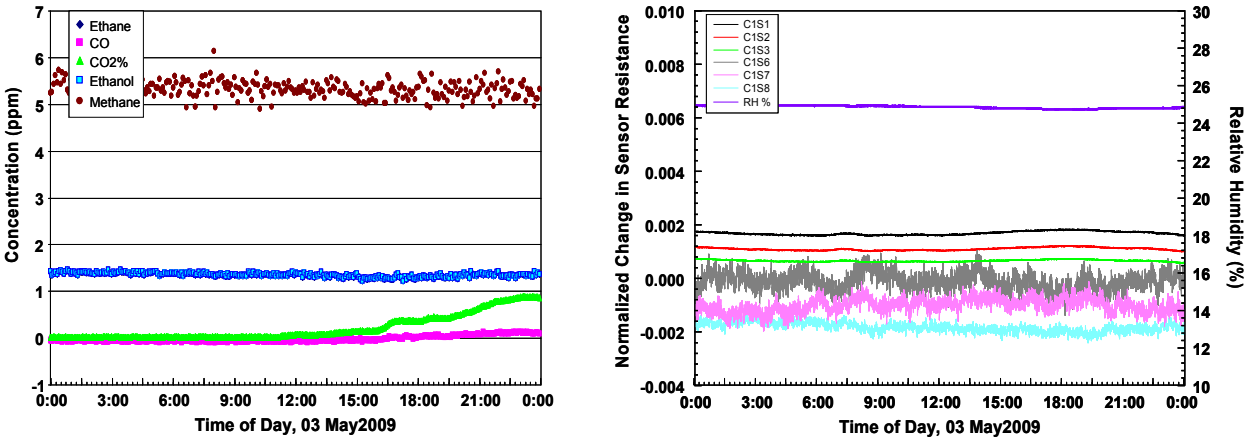

Figure 3. Data from an inactive day in the REMS. *The plots on the left show FTIR data taken on air from the REMS on a day when the door was not opened and there was no activity. The plots on the right show data from six ENose sensors and relative humidity. Individual sensor traces in ENose data have been separated and moved away from zero to aid visualization of the activity.*

In contrast, active days show significantly more variation in sensor response and in concentration of the five selected contaminants, as shown in Figure 4. The FTIR data, on left, show an increase in methane concentration from about midday on. As methane is a metabolic product, and by midday nine people had exercised in the chamber, the increase in methane is consistent with this activity. The FTIR data also show ethanol peaks in the evening, when ethanol was injected into the chamber to test the ability of the ECLSS system to remove it. The ethanol concentration fell to the background level by midday the following day. The humidity, shown in the top trace in the ENose data on the right, fluctuates significantly with activities. The ENose sensor responses follow the rise and fall of humidity.

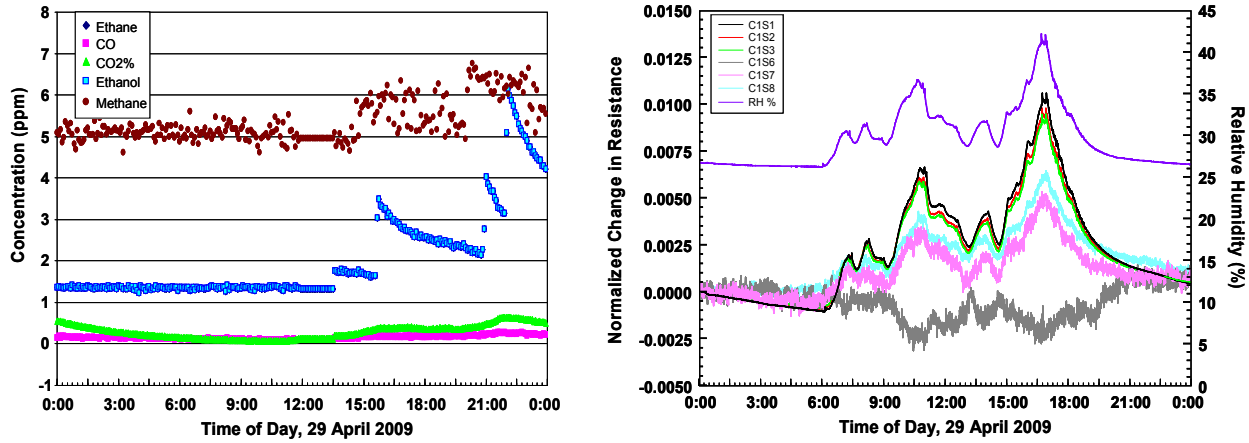

Figure 4. Data from an active day in the REMS. *The plots on the left show FTIR data taken on air from the REMS on a day when several people entered and engaged in activities. The plots on the right show data from six ENose sensors and relative humidity.*

The purpose of these experiments was to determine whether the ENose, while operating, would respond to normal activities and report false events. The FTIR analysis of REMS air and ENose sensor responses and analyses

show that REMS air never had concentrations of any of the five species measured, ethane, ethanol, carbon monoxide, carbon dioxide and methane, high enough to measure by ENose. The maximum concentration of ethane, ethanol, CO and methane was never higher than 10 ppm, and the maximum concentration of CO_2 was less than 1%. Figures 3 and 4 plots of FTIR results are indicative of concentrations detected.

Although ethanol is a target chemical species for ENose, the concentrations of ethanol injected into the REMS are significantly lower than the ENose target range, and so there is no response of ENose sensors to the presence of ethanol. For example, in Figure 4, there are several peaks in the trace for ethanol, indicating injections of ethanol into the air of the REMS. However, the maximum concentration of ethanol detected is between 6 and 7 ppm. The low end of the range for ENose detection of ethanol is 167 ppm (1/3 of the target, shown in Table 1); ENose will not detect 6 ppm of ethanol. On no occasion did the FTIR analysis detect ethanol at a concentration high enough for ENose to detect it.

During the two week period selected for detailed analysis, ENose reported ethanol several times, at concentrations ranging from 700 to 1600 ppm. 1500 ppm is the upper limit in the reportable range for ethanol, and so, generally, ethanol would not be reported by ENose for the higher concentrations. However, in addition to the two reports of 1600 ppm ethanol there were four reports of ethanol at 700-1000 ppm. In each case of a reported ethanol event above 1000 ppm (three events), Subject #9 was exercising or had just completed exercise. In the cases of three reported ethanol events of 700-800 ppm, Subject #9 was exercising once; in the other two reported ethanol events, there was no overlap as to which subjects were active. Ethanol was not reported every time Subject #9 exercised, but it was reported the majority of times.

In some, but not all, of the cases where there was ethanol reported, activities such as hand or body wash, oral hygiene, or cooking a frozen meal were reported. These were not the only times these activities were logged and there was no pattern in the timing of the activity and the ethanol report. The FTIR analysis did not find ethanol at the times that ENose reported them. These ethanol reports lead us to conclude that Subject #9 carried in a chemical species that, during exercise, released a substance which caused the ENose to respond with a false positive report of ethanol. On the two occasions that ethanol was reported but Subject #9 was not present, we must conclude that a similar substance was present on one of the other subjects.

The events reported as ethanol lasted about two hours in every case. This two hour period is longer than most exercise periods, and signifiantly longer than the reported time of 20-30 minutes for events classified as unknown. After exercise, volunteer crew members hang their shirts in the REMS to simulate the situation where moist clothing cannot be removed from ISS. Two hours is consistent with the period that moist clothing might need to dry; a substance that has been transferred to clothing and mixed with moisture would then evaporate into the REMS environment until the clothing has dried. Ethanol was never reported when there were no volunteer crew members present. The chemical species causing ENose to report false positive events of ethanol has not been identified; it could come from a number of sources.

ENose did not respond specifically to activities such as hand or body washing, oral hygiene, or preparation of meals. It is not possible to determine from ENose sensor responses when those activities occurred. In addition, while it is clear that during activity the humidity in the chamber rose and the ENose sensors responded to that rise in humidity, ENose did not mistake that rise for targeted events. Inspection of the plots of ENose sensor response shows that the rise in humidity starts daily around 7:00, and there is a decline starting around 18:00. This rise and decline in humidity corresponds to opening the chamber in the morning, the start of activity, and the last person leaving the chamber in the evening. Humidity rises and falls throughout the day; these changes in humidity correspond to opening the chamber and to various activities.

During the ENose Technology Demonstration on ISS, peaks in humidity were often found at times corresponding to scheduled exercise periods. However, the peaks in humidity could not reliably be used to determine that exercise had taken place. The peaks appeared only when there was exercise, but did not always appear during exercise. The same situation was found with ENose in the REMS. As the experiments in the REMS showed, not everyone will release sufficient water into the air during exercise to change the surrounding humidity, although some people will do so some of the time.

IV. Conclusion

Operation of ENose in the REMS showed primarily that ENose does not generally respond to daily events by reporting either unknown events or false positives. While there are certainly many odors developed in the course of daily work, the chemical species responsible for the odors are generally at too low a concentration to trigger event

recognition by ENose; for an event monitor to be effective, it must respond to the species it is looking for and not trigger unknowns for daily events. This is the situation seen with ENose.

The reports of several two hour events of ethanol show a previously unobserved issue with ENose. This is the first instance in which a false positive of ethanol has been reported by ENose. It is interesting to note that ethanol is generally considered to be the "gold standard" analyte; ENose has, until now, had a 100% success rate in detecting, identifying and quantifying ethanol within the target range. That there is a substance which will cause a false positive reponse in ENose is interesting, and we will attempt to identify it using model techniques described in an accompanying paper in this conference[9].

While only two weeks of data were analyzed in detail, once it appeared that the reported ethanol events were associated with Subject #9, logs and analysis for the rest of the month of May were checked. As with ethanol events in the two week detailed period, ethanol events lasted for about 2 hours, while other, unknown events were generally 30 minutes long. In addition, 80% of ethanol events occurred when Subject #9 was exercising or had just completed exercise. It is possible that by interviewing Subject #9 we might be able to identify the substance.

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[1] O'Rourke, M.E., Perry, J.L., and Carter, D.L., "A Water Recovery System Evolved for Exploration," *Proc. 36th International Conference on Environmental Systems*, 2006-01-2274, SAE, Warrendale PA, 2006.

[2] Carter, D.L., Tabb, D. and Perry, J.L., "Performance Assessment of the Exploration Water Recovery System," *Proc. 38th International Conference on Environmental Systems*, 2008-01-2140, SAE, Warrendale PA, 2006.

[3] Ryan, M.A., Shevade, A.V., Zhou, H. and Homer, M.L., "Polymer-Carbon-Composite Sensors for an Electronic Nose Air Quality Monitor," *MRS Bulletin*, 29, 714, 2004.

[4] Ryan, M.A., Shevade, A.V., Kisor, A.K., Manatt, K.S., Homer, M.L., Lara, L.M. and Zhou, H., "Ground Validation of the Third Generation JPL Electronic Nose," *Proc. 38th International Conference on Environmental Systems* 2008-01-2044, SAE, Warrendale PA, 2008.

[5] Ryan, M.A., Zhou, H., Buehler, M.G., Manatt, K.S., Mowrey, V.S., Jackson, S.P., *et al.*, "Monitoring Space Shuttle Air Quality Using the JPL Electronic Nose," *IEEE Sensors Journal*, 4, 337 2004.

[6] Homer, M.L., Yen, S.-P.S., Ryan, M.A., Shevade, A.V., Zhou, H., Kisor, A.K., *et al.*, "Second Generation Electronic Nose," *NASA Tech Briefs*, **31 (8)**, 63 2007.

[7] *Spacecraft Maximum Allowable Concentrations for Airborne Contaminants,* Toxicology Group, Environmental Factors Office, Habitability and Environmental Factors Division Space, Life Sciences Directorate, NASA-Lyndon B. Johnson Space Center, JSC20584, October, 2008.

[8] Ryan, M.A., Manatt, K.S., Gluck, S., Shevade, A.V., Kisor, A.K., Zhou, H., Lara, L.M. and Homer, M.L., "Operation of the Third Generation JPL Electronic Nose on the International Space Station,"; *Proc. 39th International Conference on Environmental Systems*, 2009-01-2522, SAE, Warrendale PA, 2009.

[9] Shevade, A.V., Ryan, M.A., Homer, M.L., Zhou, H., Kisor, A.K., Lara, L.M., Manatt, K.S., and Gluck, S., "Characterization of Unknown Events Observed by the 3rd Generation JPL Electronic Nose Using Sensor Response Models," *Proc. 40th International Conference on Environmental Systems,* 2010-xxxx, AIAA, Reston VA, 2010.